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PHASE RELATIONSHIPS AND REACTION SINTERING OF TRANSPARENT CUBIC ALUMINUM OXYNITRIDE SPINEL

May 1979

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ARMY MATERIALS AND MECHANICS RESEARCH CENTER
Watertown, Massachusetts 02172

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ABSTRACT

A new isobaric, condensed phase diagram in the region of stability of cubic aluminum oxynitride spinel (ALON) along the pseudobinary Al_2O_3-AlN composition join is presented, deduced primarily from various analytical measurements and microstructural observations. It is shown that cubic aluminum oxynitride spinel melts incongruently at about 2050 C and is compositionally centered at about 35.7 mole % AlN which is equivalent to the following stoichiometric composition: $Al_{23}O_{27}N_5$ or 5 AlN·9 Al_2O_3. Single-phase ALON material sintered to nearly full density exhibits transparency in visible light.

5 AlN-9 Al2O3.

INTRODUCTION

Polycrystalline aluminum oxide-based ceramics are a very important family of advanced structural ceramic materials.[1] In its pure, single-crystal form, however, aluminum oxide is an anisotropic material, showing significant directional variation in properties, such as optical and thermal expansion. As a result, true optical transparency of polycrystalline Al_2O_3 is impossible, unless all the grains are identically oriented. Further, significant strain can result at grain boundaries due to thermal expansion mismatch of misoriented grains.[2] An alternate approach to this inherent problem is the stabilization of a cubic Al_2O_3 structure. A defect cubic spinel, γ-Al_2O_3,[3] can be prepared in powder form, but fully dense ceramics have not been reported due to the ease of conversion to the more stable alpha form at moderate temperatures (∿1000 C).[4,5] However, it has been known for some time[6] that nitrogen additions to Al_2O_3 in the form of AlN can produce spinel-like structures. Since that time various efforts have been made to understand the phase equilibria in this system.[7-9] The phase diagrams do not, however, indicate the temperature limits of stability for the γ-Al_2O_3 type oxynitride material, nor has single-phase material been successfully sintered. This report describes the results of a program concerned with refining the temperature-composition stability limits of cubic aluminum oxynitride spinel (ALON – nitrogen-stabilized cubic aluminum oxide) so that fully dense, single-phase ceramics could be sintered.

EXPERIMENTAL

Sintering and phase equilibria studies were carried out in an inductively heated graphite furnace using flowing N_2 (1/2 liter per minute at about 1 atm). The starting powders of γ-Al_2O_3* (1.1 μm at 50%) and AlN* (14 μm at 50%) were ball-milled for 24 hours in alumina mills with alumina balls using an ethanol fluid media, isostatically pressed at 25,000 psi, and pre-reacted at 1200 C for 24 hours

*Cerac/Pure Inc., Butler, Wisconsin

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in gas tight flowing N_2, prior to final sintering studies. Only small amounts of impurities were picked up in the ball-milling procedure. However, more sophisticated sintering studies would require an improved mixing procedure.

Final reaction and sintering runs at elevated temperatures were carried out for one hour so that direct comparison between runs could be made. Weight loss was determined for each specimen and each was characterized by X-ray diffraction and reflected light microscopy. Density measurements and transmitted light microscopy were also carried out on selected products. Our fundamental premise is that we are attempting to deduce the high temperature equilibrium relations and sintering mechanisms from the resultant products. Without the use of sophisticated apparatus, volatility is extremely difficult to suppress, so quantitative measurements of weight loss will yield useful information on vapor phase formation. The reaction samples were contained in a covered BN crucible with a sight hole for pyrometric temperature measurement. Neutron activation analysis of the AlN indicated 1.7 wt% (~1.5 mole % Al_2O_3) oxygen. The AlN powder also contained about 1 to 2 wt% of unnitrided Al metal powder.

RESULTS AND DISCUSSION

Figure 1 illustrates the temperature-composition stability limits for cubic aluminum oxynitride spinel (ALON) in the Al_2O_3-AlN system for 1 atm of flowing N_2 gas. The phase relationships were deduced from careful analyses of both microstructural and X-ray diffraction data. The Al_2O_3-AlN system is a pseudobinary composition join in the Al-N-O system. Hence, the phase rule allows for phase fields with up to three coexisting phases. The designations 12H, 21R, and 27R refer to the so-called "AlN" polytypes.[10] In this system there seems to be an intimate relationship between liquid formation and the appearance of the various polytypes. Further, the morphology of the polytypes are variable and seem to reflect the difficulty in attainment of equilibrium. It is our conclusion from this work in the vicinity of the ALON stability field and also other parts of this system that some of the polytypes are metastable products of quenched or poorly quenched liquids. We have not yet determined how to differentiate between AlN polytypes which are metastable from those which are not. Hence, in Figure 1 we have "dashed in" all the phase boundaries dealing with polytypes and in some cases have not differentiated between a liquid and the polytypes.

There is a relatively wide range of compositional stability, roughly centered at 35.7 mole % AlN, and a maximum in thermal stability at about 2050 C. At this point ALON seems to melt incongruently into one alumina-rich, stable liquid and one nitride-rich, unstable (volatile) liquid. At about 2000 C on the AlN-rich side of the single-phase field, vaporization increases dramatically which kinetically seems to influence reactions in the single-phase region.

As previously indicated by McCauley[11] a constant anion spinel model seems to predict an ALON composition at 35.7 mole % AlN. Using the chemical formula

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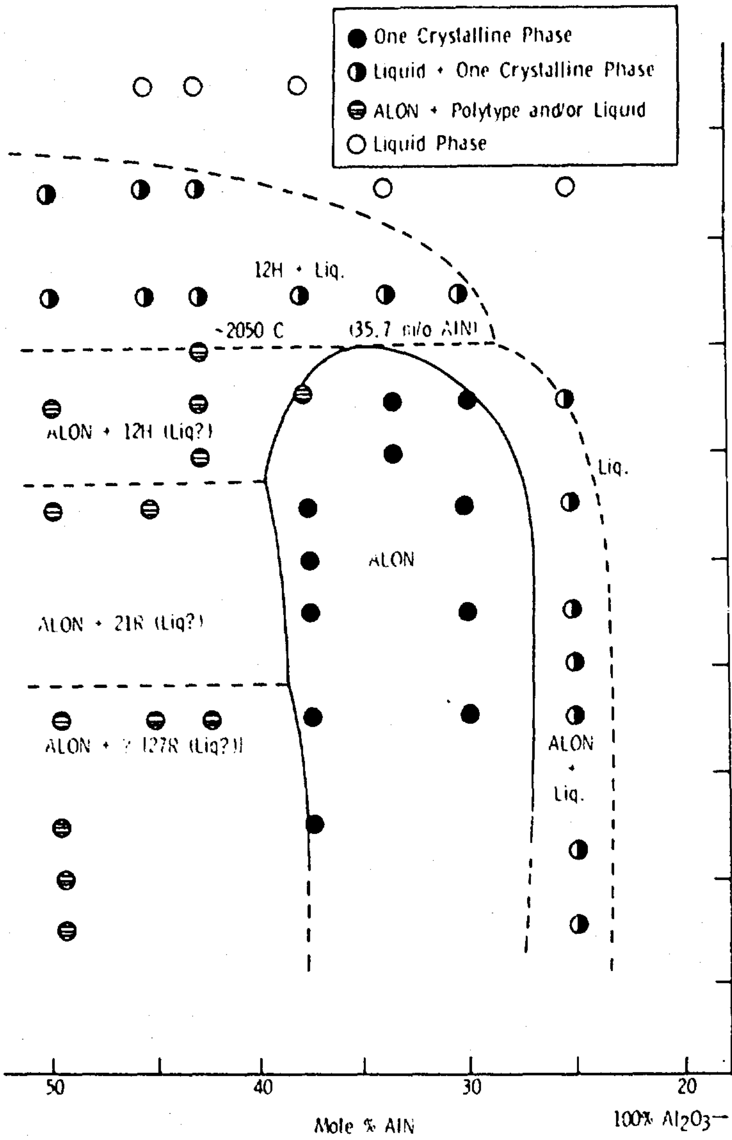


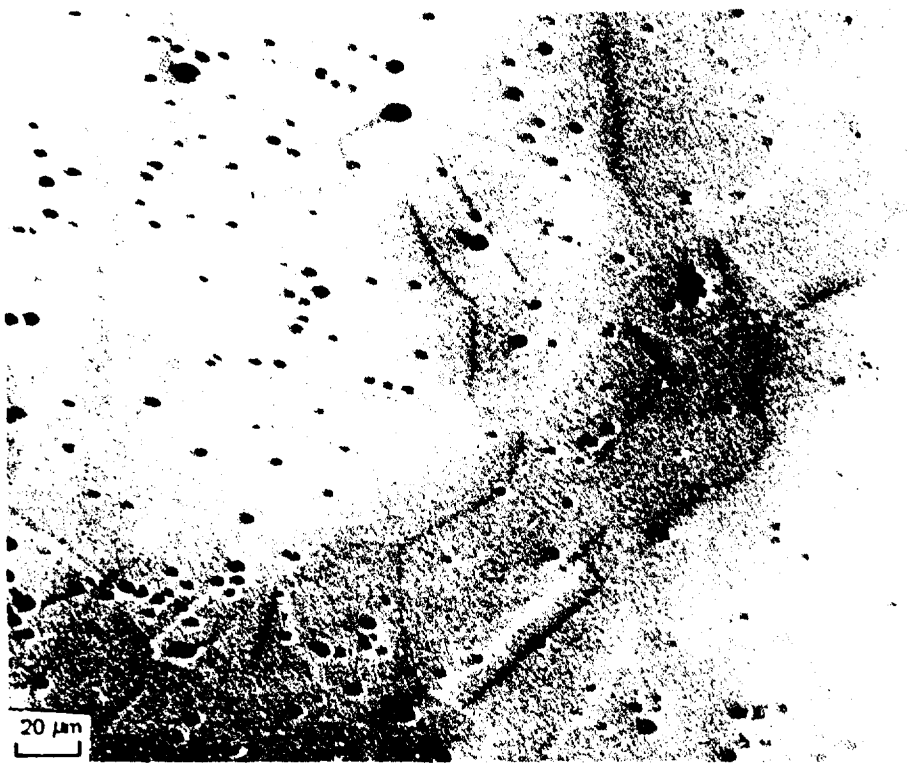
Figure 1. Proposed isobaric (1 atm of flowing N_2) high-temperature phase relationships in the region of ALON stability in the pseudobinary Al_2O_3-AlN composition join.

$Al_{(64+x)/3} \square_{(8-x)/3} O_{32-x} N_x$ obtained from this model the following composition for N = 5 can be calculated:

$$Al_{23}O_{27}N_5 \equiv 5\ AlN \cdot 9\ Al_2O_3.$$

Crystalline solution stability limits were determined by detailed reflected light microscopy and refined lattice parameters. Figure 2 illustrates the phase assemblages on the 37.5 mole % AlN composition line on either side of an apparent phase boundary line between the ALON single-phase field and the liquid plus ALON and 12H polytype region. Note the dramatic disappearance of porosity in Figure 2a and the concurrent appearance of liquid. The liquid and 12H polytype appear as the lighter colored intergranular phases in Figure 2a; in Figure 2b the darker circular areas are remnant porosity. This liquid occasionally quenches to a non-crystalline phase, but also crystallizes into various types of AlN polytypes, in this case the 12H polytype. Figure 3 illustrates the microstructure of the ALON plus liquid region on the Al_2O_3-rich side of the single-phase field.

a. 2025 C - in Liquid + ALON(SS) Field; 10.04 Wt% Loss



b. 1975 C - in ALON(SS) Field; 2.88 Wt% Loss

Figure 2. Microstructures of phase assemblages in the Al_2O_3-AlN system; 37.5 mole % AlN.

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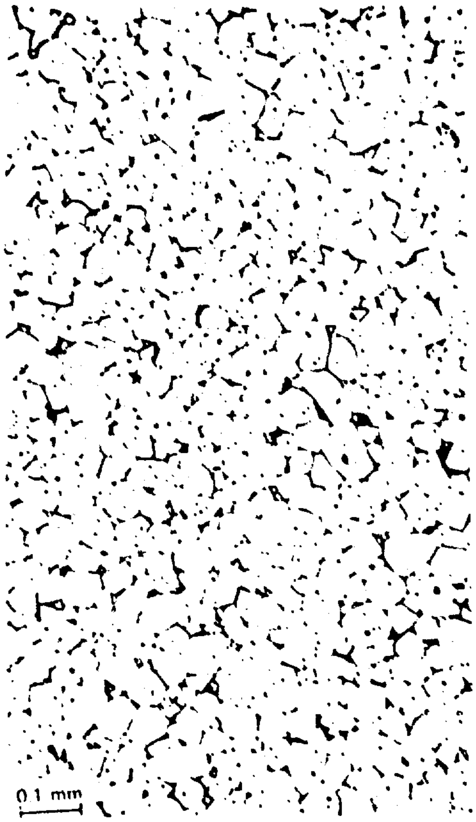
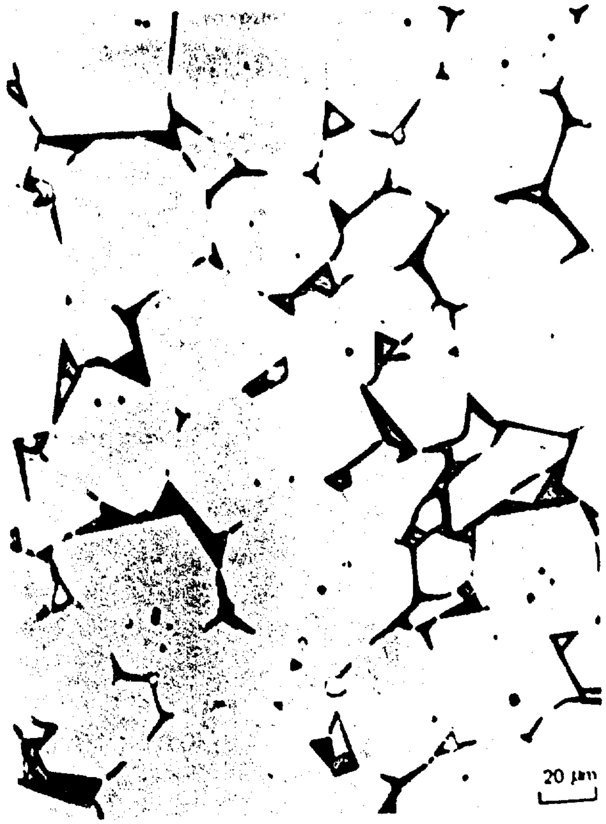
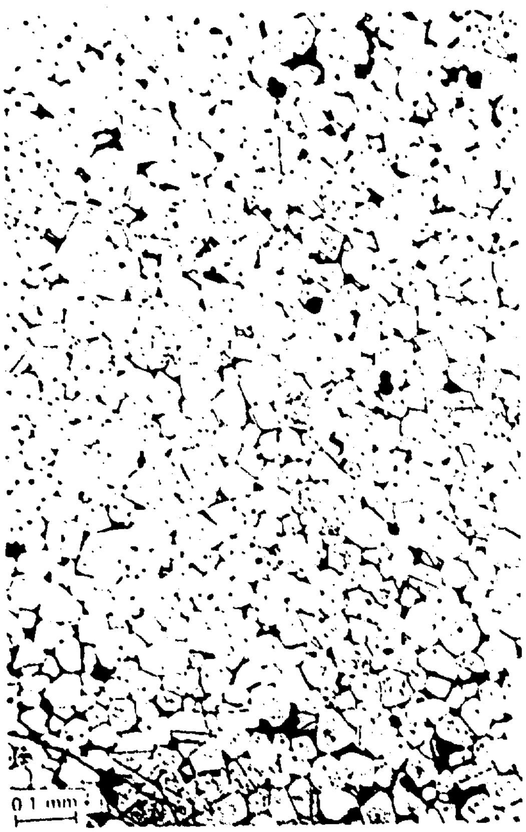


a. ALON(SS) + Liquid



b. ALON(SS) + Liquid



c. Etched

Figure 3. Microstructures of phase assemblages in the Al_2O_3-AlN system; 25 mole % AlN; T = 2025 C.
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In order to confirm the microstructurally determined range of ALON crystalline solution, refined lattice parameters were determined by a least-squares technique on X-ray powder diffraction data. These data are illustrated in Figure 4 along with those obtained by Lejus[7] on material fabricated at 1700 C. There is about a five mole % AlN difference between a least-squares line through the 1975 C data and the Lejus line. The difference can possibly be ascribed to a higher oxygen content of the latter material or the sluggish reaction rates at 1700 C. The equation for this line is indicated on the figure.

ALON ceramics of various nitrogen compositions can be sintered using the experimental conditions previously indicated to 99% of theoretical density. The theoretical densities were calculated using the constant anion model and the refined lattice parameters. Figure 5 shows a typical set of characterization data on a series of runs at 37.5 mole % AlN. By relating these data to the microstructures illustrated in Figure 2 it can be seen that the appearance of liquid results in a dramatic increase in volatility, represented by weight percent loss on the figure; percentage of theoretical density is also indicated in parentheses. A reaction and sintering scheme is also indicated on the figure.

Reaction and Sintering → Sintering (No Liquid) →

Sintering (Liquid + Vapor Formation)

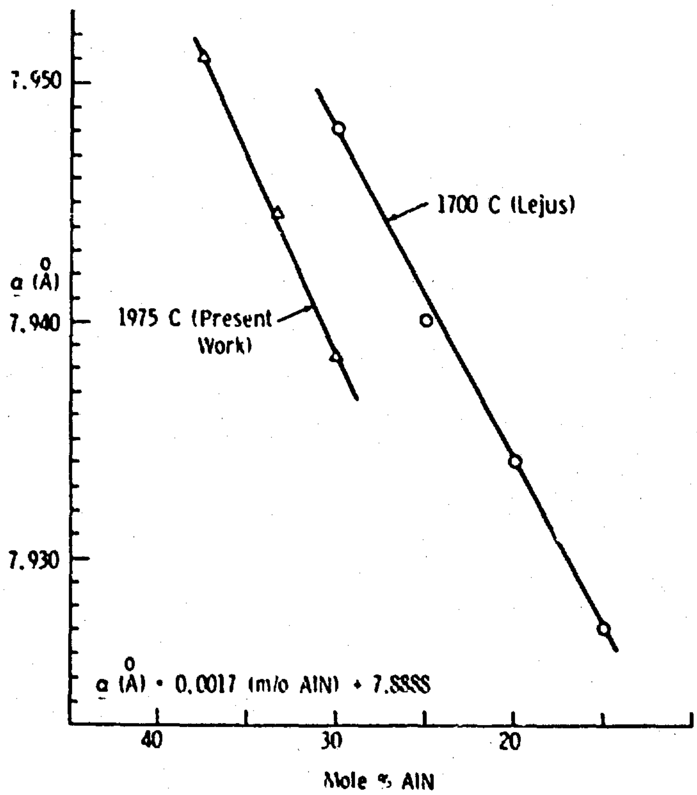

Figure 4. Refined lattice parameters of cubic aluminum oxynitride spinel (ALON) as a function of mole % AlN at 1975 C.

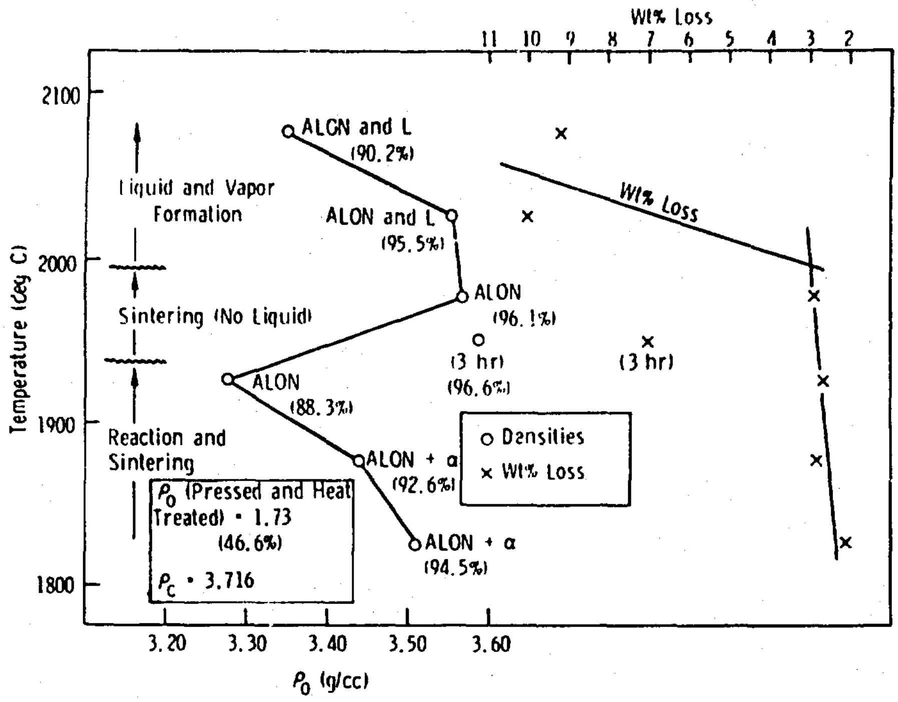

Figure 5. Variation of density and weight percent loss with temperature (1-hr runs) of 37.5 mole % AlN mixtures.

A preliminary apparent decrease in density occurs during formation of ALON, caused by the diminishing amount of the higher density α-Al_2O_3. Optimum properties and liquidless sintering must be carried out below about 2000 C to prevent the formation and subsequent vaporization of a liquid phase. Residual, quenched liquid, either as a noncrystalline phase or as one of the "AlN" polytypes, will have a large effect on the properties of the material. Note the increase in density for the material sintered for three hours.

Formation of sintered, single-phase ALON ceramics free of second phases can be easily accomplished in the single-phase field. Figure 6 illustrates a typical microstructure of material exhibiting a relatively large amount of remnant porosity. This material seems to react and sinter quite rapidly into a uniform grain size microstructure. For our experimental conditions, grain growth is quite rapid, with an average size somewhere between 50 and 100 μm. An abundance of apparent spinel-law ({111}) twins is also evident in Figure 6b, which seem to polish at different rates, since some are elevated and others are recessed. A small increase in sintering temperature (1975 C to 2025 C) results in much reduced residual porosity and a microstructure that only microscopic defocussing at low magnification (Figure 7a) will resolve the grain boundaries; scanning electron micrographs of fracture surfaces of this same material are illustrated in Figures 7b and c to further illustrate the microstructure.

Preliminary properties are now being measured on sintered ALON materials. Figure 8 illustrates a polished disk (0.035" thick) of a 30 mole % AlN material exhibiting a high degree of transparency; the word ALON is behind the disk. This same material has a hardness of up to 1850 Knoop (100 g), an elastic modulus

Figure 6. Microstructures of single-phase cubic aluminum oxynitride spinel; T   1975 C; 30 mole % AlN; t   hour.

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a. Reflected Light -- Slightly Defocussed to Bring out Microstructure

b. SEM -- Fracture Surface

c. SEM -- Fracture Surface

Figure 7. Microstructures of single phase cubic aluminum oxynitride spinel; T = 2025°C; 30 mole % AlN; t = 1 hour.

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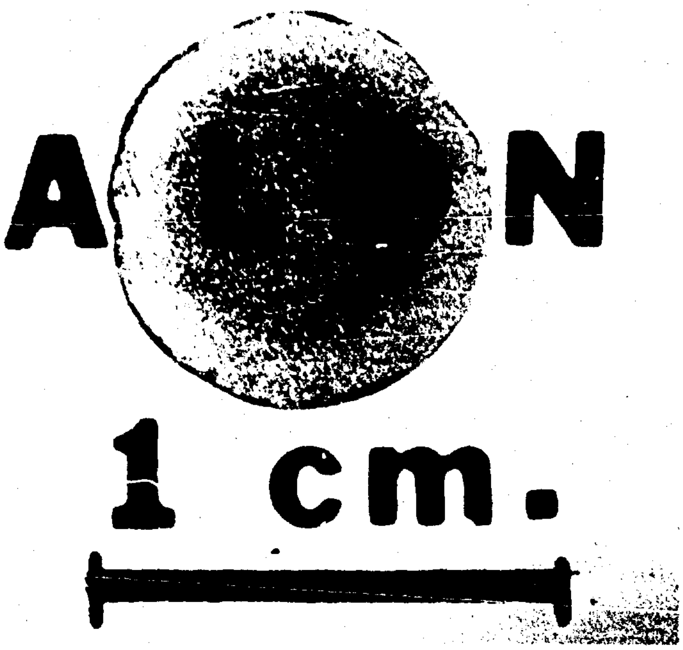

Figure 8. Transparent disk of single-phase cubic aluminum oxynitride spinel.
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(ultrasonic) of 46 × 10^6 psi, a dielectric constant at 10 MHz of 8.56, a visible light (0.55 μm) refractive index of 1.77, and an infrared cutoff of 5.20 μm. Heat treatment in air for 20 hours at 1100 C resulted in a negligible weight gain of 0.03 wt%, indicating an excellent oxidation resistance at this temperature.

SUMMARY AND CONCLUSIONS

A refined high temperature phase diagram in the region of stability of cubic aluminum oxynitride spinel (ALON) along the Al_2O_3-AlN composition join has been determined. This material can also be described as nitrogen-stabilized cubic aluminum oxide. Using this newly determined diagram, single-phase ALON has been reactively sintered to nearly full density. Sintering is carried out quite easily and polished thin disks exhibit visible light transparency. The lattice parameter of ALON varies with composition from 7.938 Å for 30 mole % AlN to 7.951 Å for 37.5 mole % AlN sintered at 1975 C. At this temperature the limit of ALON crystalline solution is from 40 to about 27 mole % AlN.

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Technical Report AMMRC TR 79-24, May 1979, 12 pp – illustrations, D/A Project 1L161102AH42, AMCMS Code 611102.H420011

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A new isobaric, condensed phase diagram in the region of stability of cubic aluminum oxynitride spinel (ALON) along the pseudobinary Al_2O_3-AlN composition join is presented, deduced primarily from various analytical measurements and microstructural observations. It is shown that cubic aluminum oxynitride spinel melts incongruently at about 2050 C and is compositionally centered at about 35.7 mole % AlN which is equivalent to the following stoichiometric composition: $Al_{23}O_{27}N_5$ or 5 AlN·9 Al_2O_3. Single-phase ALON material sintered to nearly full density exhibits transparency in visible light.

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